Pilkington / Nanofilm hybrid anti-reflection coating – White paper

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ABSTRACT: Several simple methods of imparting hydrophobicity and oleophobicity to anti-reflection (AR) coated glass are discussed. By way of example, experimental evidence is provided for a combination of Pilkington **OptiView**™ with Nanofilm UltraSEAL ABW topcoat, showing that the topcoat provides hydrophobicity and oleophobicity to the Pilkington **OptiView**™, while lowering the coefficient of friction. Additionally it has excellent resistance to UV radiation, neutral salt spray, condensation and abrasion, making it useful for touchscreen applications. The Nanofilm coating deposited on Pilkington **OptiView**™ was found to be more durable than when deposited directly on standard float glass.

Keywords: glass, anti-reflection, low friction, hydrophobic

1. Introduction

Digital signage and displays are one of the media's most powerful sources. The demand for digital signage and displays is steadily growing. Digital displays are being used in many markets and are prevalent in transportation, restaurants, hotels, schools, retail, healthcare, and financial institutions. There is an increasing desire to add additional functionality to the cover glass for such displays, including touch functionality, anti-reflection (AR) coatings, reduced friction for a "smoother" feel, anti-fingerprint (oleophobic), and so on.

Pilkington **OptiView**™ is a commercially available high performance AR product that has very thin high/low refractive index layers applied to one surface during the manufacture of the glass itself using the float process. It is available on large glass sizes up to 6000 x 3200 mm. This hard and durable coating reduces the visible reflection of the coated surface to <1% versus 4% for clear glass, as shown in Figure 1. It is also available as a dual-coated laminated version. The Nanofilm UltraSEAL ABW coating provides both hydrophobic and oleophobic properties as well as a reduction in friction. The result of this is a low maintenance/easy clean surface. An oleophobic surface typically helps to reduce the appearance of fingerprints.

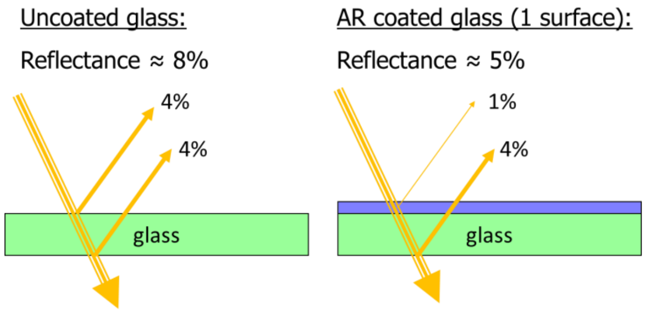

Figure 1 *– The effect of a single-sided AR coating on reflectance*

A hybrid coating allows the manufacturer to combine multiple properties in a single product, giving them wider latitude to choose the "best in class" coating for each property. High quality AR coatings are applied by depositing multiple layers, with the final layer of SiO_2 leaving the surface with an abundance of reactive Si-OH surface groups. Several chemistries are known which can form covalent ("permanent") bonds with these groups, allowing the manufacturer to tailor the surface properties with a final top layer. Because the top layer interacts with the AR layer at the molecular level, delamination, peeling, or bubbling is not an issue.

There are several options available to treat glass display surfaces with the intent of solving one or more of the following problems. High friction can be reduced by coating with a low friction material; similarly the potential of surface scratching is lessened through use of a coating to improve scratch resistance. The visibility of fingerprints is reduced using an oleophobic coating which makes them less visible and easier to clean. The most frequently used approach is to add a hydrophobic coating over the glass or AR surface. These coatings can interact with the surface in a number of ways. The first type of coating is typically a polysiloxane wax. Unfortunately, polysiloxane waxes are held to the surface only by an electrostatic attraction and the coating is easily removed after only a few cleanings.

A second class of coating is a self-assembled monolayer, usually of silane molecules for glass or SiO_2 surfaces. Silane molecules can pack together tightly, presenting a completely alkylated (or fluoroalkylated) surface to the environment. These monolayers have long been recognized for their hydrophobic properties. Additional variations within the silane molecule can impart additional properties to the coated surface such as resistance to UV degradation, improved (lowered) coefficient of friction, and enhanced oil repellency. These coatings are much thinner than wax coatings – the thickness of the coating is only equal to the length of the molecules themselves, typically in the 5 – 20 nm range. These create a conformal coating to the porous surface. Because the coatings are so thin (much less than the wavelengths of visible light), they do not change the appearance of the surface or affect the optical transmission of the AR coating (if present).

The individual molecules that make up a self-assembled monolayer are capable of forming a covalent bond to the Si-OH moieties on a silica surface. Further, the molecules are capable of forming covalent linkages between themselves, creating a dense network of bonds between the molecules, which are in turn firmly anchored to the surface. The integrity and durability of this layer is determined in part by the active ingredient in the coating and its ability to quickly form these intermolecular bonds. If the coating has a low reactivity, the molecules within it may graft to the surface at lower densities, resulting in a patchy coating, which can be more easily degraded by cleaning or the environment. The nanocoatings developed by Nanofilm are optimized for fast and complete reactivity to ensure the longest durability and best abrasion resistance.

Testing of coated glass for architectural or automotive applications is typically harsher than most display standards and this paper will describe some of these tests. Durability of displays and signage, particularly in public, is critical. As a result many display standards are perhaps not severe enough. With this in mind we have subjected the hybrid coating to architectural testing which is typically much harsher. The results are described below.

2. Experimental

Pilkington **OptiView**™ is a permanent multi-layer anti-reflection coating stack consisting of high/low refractive index layers, produced using atmospheric pressure chemical vapor deposition (AP-CVD) on a float glass line. Multiple coaters are positioned in the float bath, as shown in Figure 2, allowing the production of very large glass sheets. This coating reduces the normal reflection of a glass/air interface from 4% to less than 1%. As this is an on-line pyrolytic process, it produces highly durable hard coatings, achieving ASTM C 1376 and BS-EN1096:2012 Class A, demonstrating its suitability for use on the exterior of buildings.

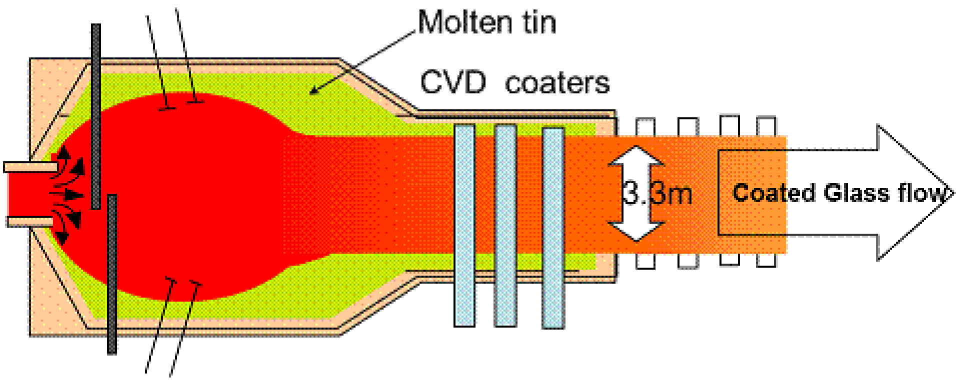

Figure 2 *– Schematic showing a float tin bath and CVD coaters*

Deposition of Nanofilm coatings on a glass substrate can be accomplished by several methods including spray or wipe on and vacuum deposition. The coating (except for the vacuum deposition method) is a low viscosity (3 – 5 cP) solution in a high flashpoint organic solvent. The coating is applied to the surface with a coverage of ~1 mL/sq. ft. at room temperature (ideal coating conditions are 5 – 33 °C and 50 – 80% RH), allowed to cure for 30 – 60 seconds, then the excess is wiped or rinsed off with hot pressurized water. The rinse water can be recycled after separation of the organic components. Additional products have been developed for application under special conditions such as glass or AR-coated plastic up to 74 °C.

Samples used in the tests described herein were coated by spraying on UltraSEAL ABW, curing at room temperature for 30 seconds, and rinsing off the excess with hot water. Coatings were deposited on standard clear float glass, and on Pilkington **OptiView**™. The subsequent testing included condensation, acid, and salt-spray resistance, as well as abrasion testing. Samples were then analyzed optically, as well as by contact angle measurements.

BS EN1096:2012 (*Glass in building — Coated Glass*) is a European standard that defines the characteristics, properties and classification of coated glass for use in domestic and commercial buildings. Part 2 covers accelerated weathering and durability testing, which was carried out as part of this study. These tests are commonly used to evaluate the resistance of the coating natural weathering conditions and physical abrasion. A sample 300 x 300 mm in size is used for each test. The tests consist of:

- Condensation resistance with the glass samples held at 40° C
- Acid resistance in an SO_2 atmosphere, cycling the temperature every 24 hours
- Neutral salt spray resistance - NaCl dissolved in water (50 g/l ± 5 g/l)
- Abrasion resistance consisting of a felt pad and 4 N load

For EN1096 Class A, where the coated surface of the glass can be positioned on the outer or the inner face of the building, the following is required:

- Condensation resistance – 21 days
- Acid resistance – 5 cycles
- Neutral salt spray resistance – 21 days
- Abrasion resistance – 500 strokes

Optical transmission measurements were carried out using a Perkin Elmer Lambda 900 spectrometer, which covers the 280-2500 nm spectral range. Additional measurements were done at 550 nm and 900 nm, using an integration time of 5 seconds to improve the signal to noise ratio, to be representative of visible and solar transmission. The tested samples were compared to a baseline sample that had not undergone durability testing. The mean was taken of six samples of each of the Nanofilm experiments. To pass the EN1096-2 standard a maximum absolute deviation after testing of 0.05 is required for the abrasion test and 0.03 for the other tests.

Water contact angle testing was carried out (which is not part of the EN1096 standard), according to NSG technology document 003R9211. The testing employed 2 µl droplets of deionised water in at least five places on the same piece and taking the mean of the diameter. Any asymmetrical drops were discarded. A solid surface is hydrophobic if the water contact angle is greater than 90°. Below 90°, the droplet forms a spherical segment and the contact angle for a known volume can be calculated based on the droplets diameter. Similarly above 90°, the droplet forms a partial sphere with a "missing" segment at the bottom and again the contact angle can be easily calculated. Oil contact angle testing was carried out at Nanofilm using jojoba oil.

The coefficient of friction for coated float glass was measured by ASTM method D1894 on a ChemInstruments AR-1000 at 6 in/min with a 2.5" sled weighing 200 g. The coated pieces were measured against uncoated glass and stainless steel. The testing was performed by Chemsultants (Mentor, OH).

Nano Scratch testing was performed by Nanovea (Irvine, CA) using a Nanovea Nano Module with a 2 micrometer radius diamond tip, initial load of 0.06 mN, final load of 200 mN, loading rate of 200 mN/min and scratching speed of 2.00 mm/min.

UltraSEAL ABW was coated onto clear float glass, and on Pilkington **OptiView™** and the pieces underwent Xenon Saylight B/B at 0.55 W/m^2 exposure testing under the conditions of standard SAE J2527 at Q-lab facilities in Florida, USA.

Haze, transmission, and water contact angles were measured initially and every 500 kJ of exposure up to 2500 kJ.

3. Results and discussion

Nanofilm UltraSEAL ABW was deposited on both Pilkington **Optifloat™** Clear and Pilkington **OptiView™**. Due to the properties of the coating, neither the appearance nor the transmission/reflection of either substrate was affected. There was a noticeable reduction in the coefficient of friction (CoF) of ABW coating on clear float glass, as shown in Table 1. Testing was done to ASTM D1894 (test conducted at Chemsultants, Inc., Mentor, OH). This lower friction surface is also thought to be easier to clean, reducing the appearance of fingerprints.

Table 1 *– Coefficients of friction (CoF)*

Sample	**Static CoF - glass**	**Dynamic CoF – glass**	**Static CoF – stainless steel**	**Dynamic CoF – stainless steel**
Uncoated glass (control)	0.34	0.28	0.28	0.26
UltraSEAL ABW	0.19	0.17	0.20	0.21

The results of Nano Scratch Testing of ABW coating on clear float glass (test conducted by Nanovea, Irvine, CA) indicated that the coated also offered some scratch protection to the glass, as shown in Table 2. This was thought to be the result of a lower coefficient of friction, giving a smoother feel to the surface.

Table 2 – Delamination force measurements

Sample	**Delamination force (mN)**
Uncoated glass (control)	27.7
UltraSEAL ABW	40.8

Nanofilm UltraSEAL ABW coatings have been shown to be highly oleophobic, as evidenced by the oil contact angle results in Table 3. The same high angle is obtained both directly on the base glass and on top of Pilkington **OptiView™**.

***Table 3** – Oil contact angles (OCA)*

Sample	Oil contact angle
Uncoated base glass (control)	15
Base glass + ABW	75
Pilkington **OptiView**™	25
Pilkington **OptiView**™ + ABW	75

BS EN1096:2012 durability testing was carried out on the UltraSEAL ABW coating on both clear float glass directly and on top of Pilkington **OptiView**™. Untreated Pilkington **OptiView**™ has previously been shown to pass this testing. For the UltraSEAL ABW coatings, visual inspection showed that the acid and condensation tests both appeared to have no effect, and the hybrid coating on Pilkington **OptiView**™ was slightly more durable in the neutral salt spray test than on clear glass. The results are shown in Table 4.

***Table 4** – Optical deviations and contact angle measurements following environmental testing*

Sample	Durability Test	550 nm dev.	900 nm dev.	Contact angle (°)
Base glass + ABW	Ref	N/A	N/A	103
Base glass + ABW	Acid resistance	0.06	0.10	93
Base glass + ABW	Condensation	0.05	0.08	101
Base glass + ABW	Neutral salt spray	0.01	0.05	36
Base glass + ABW	Abrasion	0.03	0.01	94
Pilkington **OptiView**™ + ABW	Ref	N/A	N/A	103
Pilkington **OptiView**™ + ABW	Acid resistance	0.12	0.01	96
Pilkington **OptiView**™ + ABW	Condensation	0.09	0.05	96
Pilkington **OptiView**™ + ABW	Neutral salt spray	0.01	0.03	80
Pilkington **OptiView**™ + ABW	Abrasion	0.02	0.00	95

In order to get more quantifiable results both optical transmission and water contact angles were measured. A typical value for untreated Pilkington **OptiView**™ is 40-60°. The optical transmission deviations from a reference sample were very small. However, as shown in Table 4 the contact angle results were more revealing. It is clear that the neutral salt spray appears to result in the greatest changes to the hybrid coating properties. Compared to the base glass, Pilkington **OptiView**™ was shown to provide a substrate with better salt spray resistance and therefore a more durable hybrid coating.

UV resistance of the coating was tested following SAE J2527. This is a performance-based protocol accepted by SAE International in 2008 and superseding SAE J1960, which was an instrument-specific protocol. In addition, the SAE J2527 allows for the use of daylight filters in place of the quartz/Type S boro filters on the SAE J1960 instrument. This removes the "extended UV" portion of the xenon radiation, which is not present in natural sunlight. Many automobile manufacturers have adopted the use of daylight filters as more representative of actual weathering conditions. No significant loss of transmission or water repellency were observed (as shown in Figure 3), and haze values did not increase over the duration of the test.

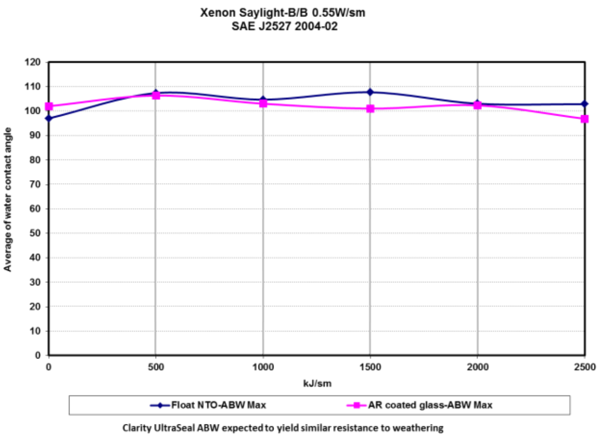

Figure 3 *- Water contact angles vs. exposure time – SAE J2527.*

Figure 4 shows the appearance of a water drop on coated glass, before and after salt spray testing. The top photo, taken before testing, clearly shows the high contact angle formed when water rests on a low surface energy surface. After testing, the coating is partially degraded, as evidenced by the lower contact angle. It is worth noting however that the surface retains a degree of hydrophobicity, indicating the coating is not completely destroyed by the test.

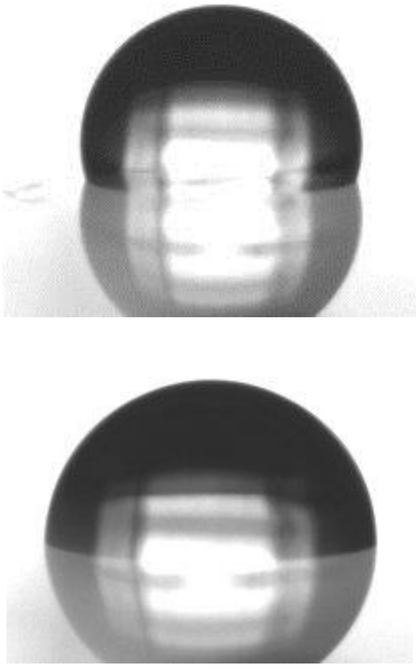

Figure 4 *- Initial hydrophobic reference sample (top) and after salt spray testing (bottom) to show contact angles. Note: images are not shown at the same magnification.*

4. Conclusions

The demand for digital signage and displays is steadily increasing. Pilkington **OptiView**™ is an excellent solution to reduce reflections from digital displays, computer screens, aircraft transparencies etc. This high performance coating reduces reflection to <1% from the coated surface, while providing high light transmittance. When combined with the Nanofilm UltraSEAL ABW coating it retains its optical properties, but gains a "smoother" feel by way of the reduced friction. In addition, the increased hydrophobicity and oleophobicity provide a surface which is lower maintenance than Pilkington **OptiView™** alone, reducing the appearance of fingerprints and making any fingerprints or other contamination easier to remove. The Nanofilm coating is also very easy to apply compared to other coating techniques. This improves the performance of the coating for touch applications. The hybrid coating also exhibits a hydrophobic surface, which means water will form beads and roll-off the surface if place vertically, rather than adhering. By combining Pilkington **OptiView**™ with Nanofilm UltraSEAL, we have created a hybrid AR glass coating that is color neutral and durable and positions Pilkington to provide manufacturers with best-in-class AR coated glass products.